

17369

DATE: October 23, 1996

4 2 0703



**START PROGRAM  
SPECIAL INVESTIGATION**

**LEAD ANALYSES OF SAMPLES FROM THE  
PETROLEUM PRODUCTS CORPORATION SITE**

**DRAFT REPORT**

Prepared by:  
Evelyn Meagher-Hartzell and Jim Rawe  
Science Applications International Corporation  
635 West Seventh Street, Suite 403  
Cincinnati, Ohio 45203

Prepared for:  
U.S. Environmental Protection Agency  
National Risk Management Research Laboratory

EPA Contract No. 68-C5-0001  
Work Assignment 1-03, Technical Directive E

Work Assignment Manager  
Mr. Edward Bates  
U.S. Environmental Protection Agency  
National Risk Management Research Laboratory  
26 West Martin Luther King Drive  
Cincinnati, OH 45268

## TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
1.0 Project Description .....	1
1.1 General Overview .....	1
1.2 Project Objective. ....	1
1.3 Experimental Design .....	1
2.0 Analytical and QA/QC Results .....	2
2.1 Groundwater. ....	2
2.2 Soil. ....	5
2.3 Sludge .....	6
2.4 LNAPL Oil .....	6
2.5 Sludge SPLP Leachate .....	6
2.6 Soil SPLP Leachate .....	7
3.0 Discussion. ....	7
Appendix A: Standard Operating Procedure for Tetraethyl Lead .....	A-1
Appendix B: Method Verification Results for Tetraethyl Lead .....	B-1
Appendix C: Example Chromatograms .....	C-1

## LIST OF TABLES

<u>Number</u>		<u>Page</u>
1	Analytical Methods .....	3
2	PPC Superfund Site Sample Results .....	3
3	Tetraethyl Lead (as Pb) MS Results .....	4
4	Lead MS Results .....	4
5	Tetraethyl Lead Laboratory Control Sample (LCS) Results .....	4
6	Tetraethyl Lead Surrogate Results: 2-Fluorobiphenyl .....	5

## **1.0 PROJECT DESCRIPTION**

### **1.1 General Overview**

The U.S. Environmental Protection Agency (EPA's) National Risk Management Research Laboratory (NRMRL) provided technical support to EPA Region IV during the analysis of contaminated soil, sludge, LNAPL oil, and groundwater samples from the Petroleum Products Corporation (PPC) Superfund Site in Pembroke Park, Florida. These samples were analyzed for total lead, total tetraethyl lead, pH, and percent moisture. The soil and sludge samples were also extracted using the Synthetic Precipitation Leaching Procedure (SPLP) and the SPLP leachates were analyzed in duplicate for lead, tetraethyl lead, and pH. Analytical results were obtained to support a possible revised risk assessment.

Technical Support was provided under EPA-NRMRL's Superfund Technical Assistance Response Team (START) Program. EPA-NRMRL's contractor, Science Applications International Corporation (SAIC), coordinated sample analysis. DataChem Laboratories, SAIC's subcontractor laboratory, analyzed the samples.

### **1.2 Project Objective**

The objective of the project was to analyze PPC soil, sludge, groundwater, LNAPL oil, soil SPLP leachate, and sludge SPLP leachate samples for lead, tetraethyl lead, pH, and percent moisture (soil and sludge only).

### **1.3 Experimental Design**

#### **1.3.1 Sample Collection/Mixing**

Soil, sludge, groundwater, and LNAPL oil samples were received at the laboratory in July and August 1996. To ensure that representative soil and sludge sub-samples were obtained, SAIC separately mixed and sub-sampled the two 5-gallon buckets containing the soil and sludge prior to analysis. The groundwater and LNAPL oil samples did not need to be mixed or sub-sampled prior to analysis.

#### **1.3.2 Sample Analysis**

Soil, sludge, groundwater, and LNAPL oil samples were analyzed for lead, tetraethyl lead, and pH; the soil, sludge, LNAPL oil, and one groundwater sample were analyzed in duplicate. SPLP leachates from sludge and soil samples were also analyzed in duplicate for lead, tetraethyl lead, and pH. The samples were prepared, extracted, and analyzed according to the procedures outlined in Table 1.

Tetraethyl lead and lead matrix spikes (MSs) were performed on the soil, sludge, LNAPL oil, one groundwater sample, and one SPLP leachate (i.e., the leachate obtained from the soil sample). Tetraethyl lead laboratory control samples (LCSs) were analyzed for both solid and liquid matrices. A surrogate (e.g., 2-fluorobiphenyl) was also added to soil, sludge, groundwater, and leachate samples during tetraethyl lead analyses; 2-fluorobiphenyl concentrations were measured and reported during each primary and QA/QC analysis of the samples.

### **1.3.3 Preliminary Screening/Method Verification**

No standard method is currently available for analyzing tetraethyl lead in the various matrices of concern for this study. As noted in Table 1, a non-standard GC/FID method (based on NIOSH 2533) was used to quantitate tetraethyl lead (see Appendix A). This method uses various solvents (e.g., hexane, methylene chloride, and 50/50 methylene chloride/acetone) to extract tetraethyl lead from the different matrices, and then GC/FID to quantitate the tetraethyl lead. Prior to sample analysis, DataChem performed a preliminary screening of the soil sample to evaluate any matrix effects which may have prohibited quantitation of tetraethyl lead (e.g., significant hydrocarbon contamination). This was done by shaking the soil sample with solvent and injecting the extract into the GC and determining if the tetraethyl lead peak could be isolated. After it appeared that the tetraethyl lead could be isolated, DataChem performed a method verification study as described in Appendix B. Method verification results were used to define accuracy, precision, and detection limits for the method. This information was also used to develop and finalize a Standard Operating Procedure (SOP) detailing the procedures to used during sample analysis (see Appendix A).

## **2.0 ANALYTICAL AND QA/QC RESULTS**

Table 2 contains the lead (total and SPLP), tetraethyl lead (total and SPLP), pH, and moisture results for the PPC samples. Tables 3, 4, 5, and 6 contain associated quality control results. Groundwater, soil, sludge, LNAPL oil, and SPLP leachate (soil and sludge) results are discussed in detail in Sections 2.1 through 2.6, respectively.

### **2.1 Groundwater**

As shown in Table 2, total lead concentrations in groundwater were 65 ug/L in PMW16A and 402 ug/L in PMW05A; total lead concentrations averaged 400 ug/L in PMW05A. Tetraethyl lead concentrations in both groundwaters were less than 16 ug/L (as Pb). The pH of PMW16A averaged 1.32; the pH of PMW05A average 1.54.

**Table 1 Analytical Methods**

Matrix	Analytical Parameter	Analytical Methods <sup>a</sup>
Soil, Sludge	Pb	3051/6010A
	Tetraethyl Pb	See SOP <sup>b</sup>
	pH	9045C
	SPLP	1312
	Moisture	ASTM D 2216
LNAPL Oil	Pb	3051/6010A
	Tetraethyl Pb	See SOP <sup>b</sup>
	pH	9045C
Groundwater, SPLP Leachate	Pb	3015/6010A
	Tetraethyl Pb	See SOP <sup>b</sup>
	pH	9040B

<sup>a</sup> Test Methods for Evaluating Solid Waste, USEPA 1987, SW-846, 3rd Ed. (through Update II, November 1994), unless noted otherwise.

<sup>b</sup> See Appendix A.

**Table 2. PPC Superfund Site Sample Results**

	Lead	Tetraethyl Lead (as Lead)	pH	% Moisture
Groundwater				
PMW16A	65 ug/L	< 16 ug/L	1.32	NA
PMW16A Dup	NA	< 16 ug/L	1.31	NA
PMW05A	402 ug/L	< 16 ug/L	1.54	NA
PMW05A Dup	397 ug/L	NA	1.53	NA
SPLP Leachate - Soil				
PPC-SO-01	360 ug/L	< 16 ug/L	5.53	NA
PPC-SO-01 Dup	365 ug/L	< 16 ug/L	5.47	NA
SPLP Leachate -Sludge				
PPC-SLUDGE	2,830 ug/L	38 ug/L	1.77	NA
PPC-SLUDGE Dup	2,830 ug/L	33 ug/L	1.76	NA
Soil <sup>a</sup>				
PPC-SO-01	7,040 mg/Kg	< 3.2 mg/Kg	4.05	22.0
PPC-SO-01 Dup	7,540 mg/Kg	< 3.2 mg/Kg	2.33	NA
Sludge <sup>a</sup>				
PPC-SLUDGE	3,620 mg/Kg	< 6.4 mg/Kg	0.46	18.4
PPC-SLUDGE Dup	5,320 mg/Kg	< 6.4 mg/Kg	0.47	NA
LNAPL Oil				
PP-2	91.6 mg/Kg	1,700 mg/Kg	6.57	NA
PP-2 Dup	98.0 mg/Kg	1,710 mg/Kg	6.64	NA

NA Not Analyzed

<sup>a</sup> Sample results have not been corrected for moisture content. Some of the loss observed in the moisture analysis may be attributed to the loss of volatile hydrocarbons.

**Table 3. Tetraethyl Lead (as Pb) MS Results**

	Sample Result	Spike Added (as lead)	Spiked Result	% Recovery
Groundwater				
PMW16A	ND (16) ug/L	640 ug/L	416 ug/L	65.0
SPLP Leachate - Soil				
PPC-SO-01	ND (16) ug/L	640 ug/L	560 ug/L	87.5
Soil				
PPC-SO-01	ND (3.2) mg/Kg	640 mg/Kg	79.4 mg/Kg	12.4
Sludge				
PPC-SLUDGE	ND (6.4) mg/Kg	640 mg/Kg	ND (3.2) mg/Kg	0
LNAPL Oil				
PP-2	1,700 mg/Kg	5,295 mg/Kg	7,130 mg/Kg	103

ND Not detected at the levels in parentheses.

**Table 4. Lead MS Results**

	Sample Result	Spike Added	Spiked Result	% Recovery
Groundwater				
PMW05A	402 ug/L	1,000 ug/L	1,440 ug/L	104
SPLP Leachate - Soil				
PPC-SO-01	360 ug/L	11,100 ug/L	10,900 ug/L	95
SPLP Leachate - Sludge				
PPC-SLUDGE	2,830 ug/L	11,100 ug/L	14,600 ug/L	106
Soil				
PPC-SO-01	7,040 mg/Kg	10,000 mg/Kg	17,700 mg/Kg	107
Sludge				
PPC-SLUDGE	3,620 mg/Kg	10,000 mg/Kg	13,800 mg/Kg	102
LNAPL Oil				
PP-2	91.6 mg/Kg	20,000 mg/Kg	19,100 mg/Kg	95

**Table 5. Tetraethyl Lead Laboratory Control Sample (LCS) Results**

	Spike Added	Spiked Result	% Recovery
Groundwater/Leachate	640 ug/L	409 ug/L	63.9
Sludge/Soil	640 mg/Kg	593 mg/Kg	92.7

Table 6. Tetraethyl Lead Surrogate Results: 2-Fluorobiphenyl

	Spike Added	Spiked Result	% Recovery
Groundwater			
PMW16A	1,000 ug/L	780 ug/L	78.0
PMW16A Dup	1,000 ug/L	705 ug/L	70.5
PMW16A MS	1,000 ug/L	780 ug/L	78.0
PMW05A	1,000 ug/L	845 ug/L	84.5
SPLP Leachate - Soil			
PPC-SO-01	1,000 ug/L	880 ug/L	88.0
PPC-SO-01 Dup	1,000 ug/L	955 ug/L	95.5
PPC-SO-01 MS	1,000 ug/L	950 ug/L	95.0
SPLP Leachate -Sludge			
PPC-SLUDGE	1,000 ug/L	1,040 ug/L	104
PPC-SLUDGE Dup	1,000 ug/L	985 ug/L	98.5
Soil			
PPC-SO-01	20 mg/Kg	30.2 mg/Kg	151
PPC-SO-01 Dup	20 mg/Kg	27.4 mg/Kg	137
PPC-SO-01 MS	20 mg/Kg	32.5 mg/Kg	163
Sludge			
PPC-SLUDGE	20 mg/Kg	20.4 mg/Kg	102
PPC-SLUDGE Dup	20 mg/Kg	25.1 mg/Kg	126
PPC-SLUDGE MS	20 mg/Kg	26.1 mg/Kg	131

Tetraethyl lead and lead MS recoveries were 65 and 104 percent, respectively. Tetraethyl lead LCS recovery was 63.9 percent, and 2-fluorobiphenyl surrogate results ranged from 78 to 84.5 percent.

## 2.2 Soil

Lead and tetraethyl lead (as Pb) concentrations in the soil averaged 7,290 mg/Kg and less than 3.2 mg/Kg, respectively. The soil's pH averaged 3.19 and the percent moisture in the soil was 22.0 percent.

The MS recovery for lead was 107 percent. The LCS recovery for tetraethyl lead was 92.7 percent. The MS recovery for tetraethyl lead, however, was only 12.4 percent and 2-fluorobiphenyl surrogate recoveries were somewhat high, ranging from 137 to 163 percent. The low MS recovery may be due to matrix interferences. As a result, tetraethyl lead results in the soil may not be representative of true contaminant concentrations. Additional analyses are necessary to verify the absence of tetraethyl lead in the soil sample.



### 2.3 Sludge

Lead and tetraethyl lead (as Pb) concentrations in the sludge averaged 4,470 mg/Kg and less than 6.4 mg/Kg, respectively. The sludge's pH averaged 0.46 and the percent moisture was 18.4 percent.

The MS recovery for lead was 102 percent. The LCS recovery for tetraethyl lead was 92.7 percent. 2-Fluorobiphenyl surrogate recoveries ranged from 102 to 131 percent. However, no recovery of the MS was obtained during sample analysis. Because matrix interferences are suspected, tetraethyl lead results in the sludge may not be representative of true contaminant concentrations. Additional analyses are necessary to verify the absence of tetraethyl lead in the sludge sample.

### 2.4 LNAPL Oil

Lead and tetraethyl lead (as Pb) concentrations in the LNAPL oil averaged 94.8 mg/Kg and 1,700 mg/Kg, respectively. The oil's pH averaged 6.60.

The discrepancy between total lead and tetraethyl lead (as Pb) concentrations measured during LNAPL oil analyses appears to indicate that non-representative results have been obtained. It is possible, that despite the fact that an MS recovery of 103 percent was obtained, the unspiked peak that eluted at the tetraethyl lead retention time may not actually be tetraethyl lead (see the chromatograms in Appendix C which demonstrate significant matrix interferences). Again, because matrix interferences are suspected, tetraethyl lead results in the sludge may not be representative of true contaminant concentrations. Additional analyses are necessary to verify the concentration of tetraethyl lead in the oil matrix.

### 2.5 Sludge SPLP Leachate

Lead and tetraethyl lead (as Pb) concentrations in the SPLP leachate obtained from the PPC sludge averaged 2,830 ug/L and 36 ug/L, respectively. The sludge SPLP leachate pH averaged was 1.76.

The lead MS recovery was 106 percent. No MS was performed for tetraethyl lead for the sludge leachate. However, the tetraethyl lead LCS was 63.9 percent, and surrogate recoveries were 104 and 98.5 for the duplicate samples.

## 2.6 Soil SPLP Leachate

Lead and tetraethyl lead (as Pb) concentrations in the SPLP leachate obtained from the PPC soil averaged 362 ug/L and less than 16 ug/L, respectively. The soil SPLP leachate pH averaged 5.50.

The lead MS recovery was 95 percent. The tetraethyl lead LCS was 63.9 percent and the MS recovery was 87.5 percent. Surrogate recoveries for the tetraethyl lead analyses ranged from 88.0 to 95.5 percent.

## 3.0 DISCUSSION

The QC results generated support the lead results for all matrices, as well as the tetraethyl lead (as Pb) results for the groundwater and SPLP leachates. However, matrix interferences were identified during analyses of the soil, sludge, and LNAPL oil samples. Since tetraethyl lead results in these media may not be representative of actual contaminant concentrations, these data should be used cautiously.

To confirm the absence of tetraethyl lead in the soil and sludge samples, a smaller sample size could be extracted (1 gram rather than 10 grams) to minimize matrix interferences, in addition to the post-extraction dilution performed. This would increase detection limits by a factor of 10 (to approximately 50-100 mg/Kg), but increased recovery of a spike would provide greater assurance that tetraethyl lead is not present at the detection limit. Some possible options for confirming the presence or absence of tetraethyl lead in the oil include an extract cleanup procedure (e.g., acid cleanup), the use of a second GC column, or the use of GC/MS.

4 2 0709

**APPENDIX A**

**Standard Operating Procedure**

**for**

**Tetraethyl Lead**

## STANDARD OPERATING PROCEDURE

for

### TETRAETHYL LEAD

#### 1.0 Scope and Application

- 1.1 This procedure is applicable to the analysis of groundwaters, SPLP leachates, soils, sludges, and oil samples for tetraethyl lead (as Pb). Approximate practical quantitation limits (PQLs) are listed below:

MATRIX	PQL
Groundwater, SPLP Leachate	25 ug/L
Soil, Sludge	0.5 ug/g
Oil	5 ug/g

- 1.2 This procedure was prepared for Technical Directive E for Work Assignment 1-03 under Contract 68-C5-0001.

#### 2.0 Method Summary

Samples are extracted with solvent and analyzed using GC/FID. The GC/FID is calibrated with a series of standards containing known concentrations of tetraethyl lead (as tetraethyl Pb). Sample extracts are injected and quantitated against this calibration curve. Results are reported as tetraethyl lead (as Pb).

#### 3.0 Health & Safety

Tetraethyl lead is extremely poisonous. Acute or chronic poisoning may occur if inhaled or absorbed through skin.

#### 4.0 Sample Preservation, Containers, Handling, and Storage

- 4.1 All samples should be stored in glass containers at 4 degrees C.
- 4.2 Groundwater and SPLP leachate samples should be extracted within 7 days of sample collection. The extracts should be analyzed as soon as possible (not to exceed 14 days).
- Soil, sludge, and oil samples should be extracted within 14 days of sample collection. The extracts should be analyzed as soon as possible (not to exceed 14 days).

#### 5.0 Interferences and Potential Problems

If peak interference problems are encountered, the chromatographic column or separation conditions may be changed.

## 6.0 Equipment/Apparatus

- 6.1 Gas Chromatograph (GC) with Flame Ionization Detector (FID) (e.g., HP-5890 GC/FID)
- 6.2 Chromatographic Column (e.g., 30 meter by 0.53 mm DB-1 or equivalent)
- 6.3 Soxhlet Extractor Apparatus and associated accessories
- 6.4 Separatory Funnel
- 6.5 Concentrator Apparatus (e.g., Kuderna-Danish)
- 6.6 Miscellaneous Equipment (volumetric flasks, vials, pipets, etc.)

## 7.0 Reagents

- 7.1 Tetraethyl lead standard (purchased from Aldrich or equivalent)
  - 7.1.1 Stock Standard with a tetraethyl lead concentration of 8.265 mg/mL (5.295 mg/mL as Pb). This standard is prepared by adding 5  $\mu$ L of tetraethyl lead to 1 mL of reagent grade hexane.
  - 7.1.2 Calibration Standards at five concentration levels bracketing the linear range of the instrument, with the low standard at a level 3-5 times the estimated method detection limit will be prepared as follows:  
  
    - STD 1: 10  $\mu$ L stock in 1 mL hexane = 52.95  $\mu$ g/mL
    - STD 2: 2  $\mu$ L stock in 1 mL hexane = 10.59  $\mu$ g/mL
    - STD 3: 1  $\mu$ L stock in 1 mL hexane = 5.295  $\mu$ g/mL
    - STD 4: 20  $\mu$ L STD 1 in 1 mL hexane = 1.059  $\mu$ g/mL
    - STD 5: 5  $\mu$ L STD 1 in 1 mL hexane = 0.265  $\mu$ g/mL
- 7.2 Hexane (reagent grade)
- 7.3 Methylene chloride (reagent grade)
- 7.4 Acetone (reagent grade)
- 7.5 1:1 methylene chloride/acetone

## 8.0 Procedure

- 8.1 Sample Extraction
  - 8.1.1 Water samples - Extract 200 mL of sample as described in SW-846 Method 3510B using methylene chloride as the extraction solvent.
  - 8.1.2 Soil/Sludge samples - Extract 10g sample as described in SW-846 Method 3540B using 1:1 methylene chloride/acetone as the extraction solvent.

8.1.3 Oil samples - Extract 1g sample as described in SW-846 Method 3580B using hexane as the extraction solvent.

## 8.2 Instrument Setup

### 8.2.1 Temperature

Injector: 225°C

FID: 250°C

Column: 120°C x 1 minute; 20°C/minute to 180°C x 1 minute

### 8.2.2 Carrier gas: Helium

### 8.2.3 Injection volume: 1 uL

## 8.3 Calibration and Analysis

8.3.1 Calibrate daily with the calibration standards in Section 7.1.2.

8.3.2 Prepare calibration graph [peak area vs. ug tetraethyl lead (as Pb)]. The relative standard deviation (RSD) among standards must be <20%.

8.3.3 Analyze samples (including QC samples).

8.3.4 Continuing calibration must be done after every 10 samples and at the end of a run using a midpoint calibration standard. The percent difference from the initial curve must be <15%.

## 9.0 Calculations

### 9.1 Water samples

Tetraethyl lead, as Pb, ug/L =  $\frac{\text{ug from curve (as Pb)} \times \text{vol. of extract, uL}}{\text{vol. injected, uL} \times \text{amt. extracted, L}}$

### 9.2 Soil/Sludge/Oil samples

Tetraethyl lead, as Pb, ug/g =  $\frac{\text{ug from curve (as Pb)} \times \text{vol. of extract, uL}}{\text{vol. injected, uL} \times \text{amt. extracted, g}}$

Note: Soil samples should be reported on a dry weight basis.

## 10.0 Quality Assurance/Quality Control

10.1 A method blank must be extracted and analyzed with every sample batch.

10.2 A laboratory control sample (LCS) must be analyzed with every sample batch.

- 10.2 One matrix spike (MS) should be extracted and analyzed with every sample batch. Spiking levels will be based on screening activities performed.
- 10.3 All soil, sludge, and oil samples will be analyzed in duplicate. For groundwaters, one duplicate analysis will be performed with each batch of samples.

#### 11.0 References

- 11.1 NIOSH Manual of Analytical Methods, Method 2533, Fourth Edition, 8/15/94.
- 11.2 USEPA Test Methods for Evaluating Solid Waste, SW-846, Third Edition through Update II, Methods 3510B, 3540B, and 3580B, January 1995.

4 2 0712

## **APPENDIX B**

### **Method Verification Results for Tetraethyl Lead**



## METHOD VERIFICATION RESULTS

for

### TETRAETHYL LEAD

EPA Contract No. 68-C5-0001, WA 1-03, TD E  
SAIC Project No. 01-0832-07-6403-X05

#### Introduction

EPA requested that SAIC coordinate the analyses of several samples for tetraethyl lead. Sample matrices included soil, sludge, SPLP leachates, groundwater, and oil. Because no standard method was available to analyze tetraethyl lead in these matrices, a modification of an air method was proposed (NIOSH 2533) and a draft standard operating procedure (SOP) was prepared (see Attachment A). To ensure that acceptable accuracy, precision, and detection limits can be obtained using this procedure, method verification testing was needed. Accordingly, Method verification testing was performed by DataChem in August 1996. Procedures and results are summarized below.

#### Procedures

- As presented in the SOP, SAIC plans to have their analytical laboratory extract analyze the soil and sludge samples using Method 3540B/GC and SPLP leachates and groundwater samples using Method 3510B/GC. Method verification testing of these methods was performed prior to sample analysis to ensure method acceptability. Because the oil sample only needs to be diluted as described in Method 3580B, separate verification testing was not needed for this matrix.

#### Soil/Sludge

Three 10 g portions of organic-free sand were spiked with tetraethyl lead at approximately 10 times the estimated practical quantitation limit (e.g., 3.096 ug/g), as determined during development of the calibration curve. The three portions of spiked sand were extracted with methylene chloride/acetone using EPA Method 3540 (Soxhlet extraction). Each sample was concentrated to a final volume of 10 mL in hexane and analyzed by GC/FID as described in the SOP.

#### SPLP Leachates/Groundwaters

Three 200 mL aliquots of reagent water were spiked with tetraethyl lead at a concentration approximately 10 times the estimated practical quantitation limit (e.g., 154.8 ug/L), as determined above. The three aliquots of spiked reagent water were extracted with methylene chloride using EPA Method 3510 (separatory funnel extraction). Each sample was taken to a final volume of 10 mL in hexane and analyzed by GC/FID as described in the SOP.

## Results

The following table summarizes tetraethyl lead spiking concentrations, tetraethyl lead recoveries, and the relative standard deviation (RSD) of the recoveries. Estimated PQLs are also included in this table.

### Method Verification Summary

Sample	Spiked Conc.	Analytical Conc.	% Recovery
Soil Spike 1	3.096 ug/g	1.412 ug/g	45.6
Soil Spike 2	3.096 ug/g	2.005 ug/g	65.8
Soil Spike 3	3.096 ug/g	1.709 ug/g	55.2
		AVG =	55.2
		RSD =	17.4
		PQL* =	0.5 ug/g
Water Spike 1	154.8 ug/L	128.0 ug/L	82.7
Water Spike 2	154.8 ug/L	119.0 ug/L	76.9
Water Spike 3	154.8 ug/L	127.5 ug/L	82.4
		AVG =	80.6
		RSD =	4.05
		PQL* =	25 ug/L

AVG = Average

RSD = Relative Standard Deviation

PQL = Practical Quantitation Limit

\* PQLs were estimated using the response of the lowest calibration standard. The PQL developed using water spike results is applicable to groundwater and SPLP leachate samples. The PQL developed using soil spike results is applicable to soil and sludge samples.

4 2 0714

## **APPENDIX C**

### **Example Chromatograms**

## Chromatogram

4 2 0715

Sample Name : std 92.35

FileName : c:\2700\data\0930046.raw

id : gca.ins

Start Time : 0.00 min

Scale Factor : 0.0

End Time : 3.50 min

Plot Offset : 0 mV

Sample #: 16

Date : 10/1/95 11:13 AM

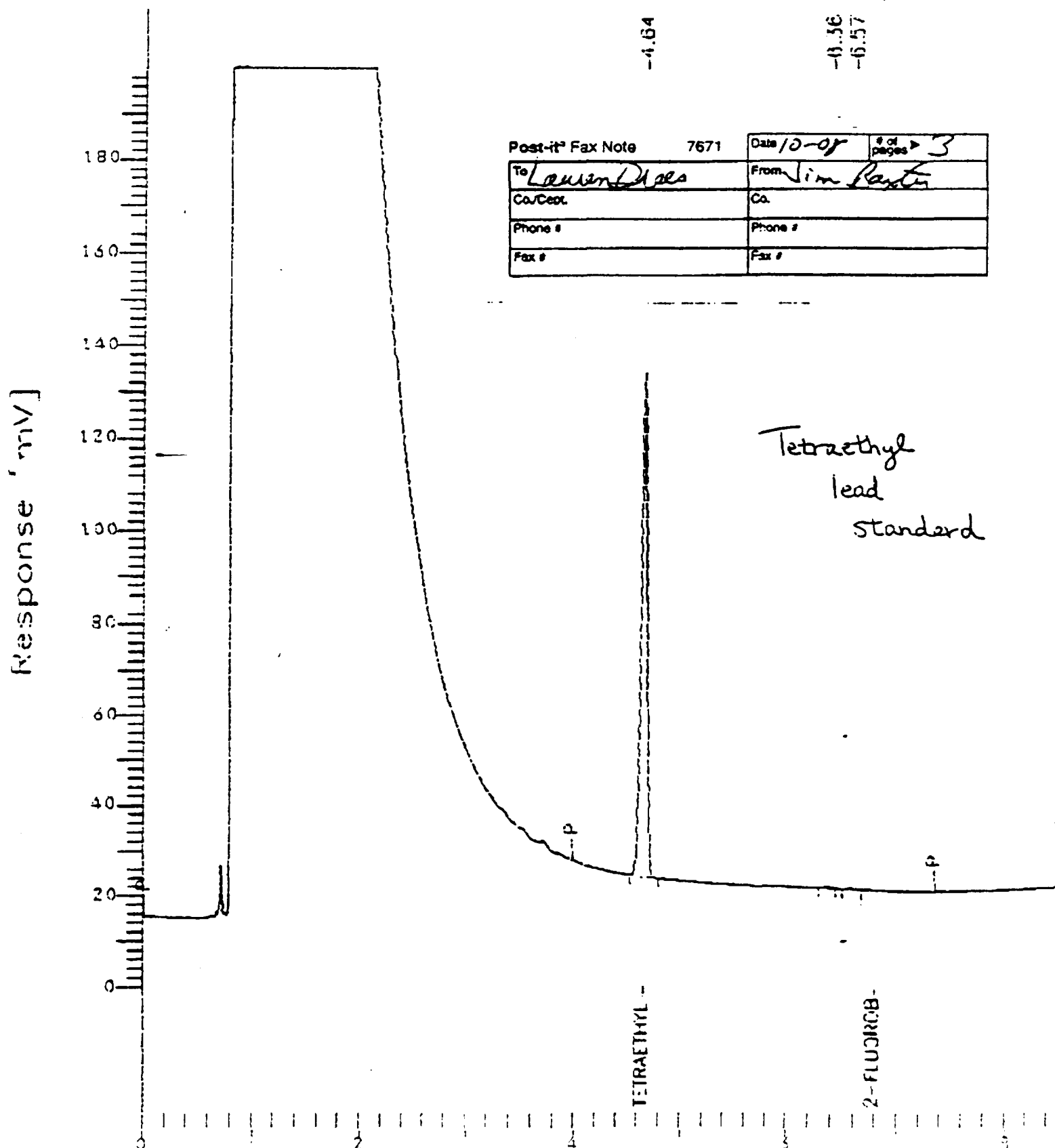
Time of Injection: 10/1/95 11:04 AM

Low Point : 0.00 mV

Plot Scale: 200.0 mV

Page 1 of 1

High Point : 200.00 mV



Chromatogram

Sample Name : 96-51380 111 11100 SUP  
File Name : C:\2700\N\inst\0950039.raw  
Method : GC/MS  
Start Time : 01:00 min  
Scale Factor : 0.0

End Time : 3.50 min  
Plot Offset : 0 mV

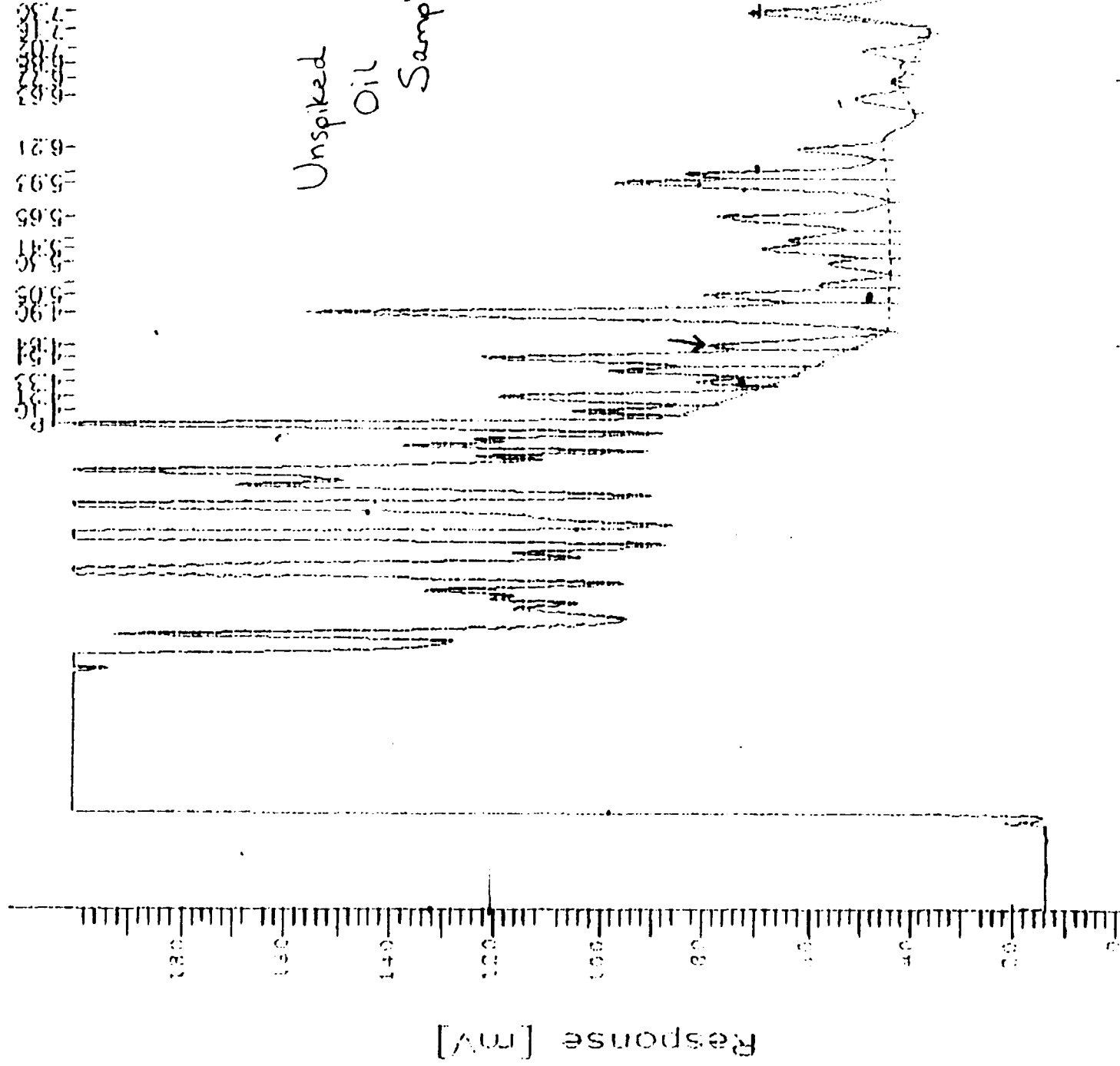
Sample #: 37  
Date : 10/1/95 09:18 AM  
Time of Injection: 10/1/95  
Low Point : 0.00 mV  
High Point : 200.00 mV  
Plot Scale: 200.0 mV

Page : of :

09:07 AM

High Point : 200.00 mV

Plot Scale: 200.0 mV



11/10/95

2-1103808-

